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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IV COPPER AND ITS ALLOYS AND THEIR EXAMINATION

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CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IV COPPER AND ITS ALLOYS AND THEIR EXAMINATION

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Indian Standard

CODE OF PRACTICE FOR PREPARATION OF METALLOGRAPHIC SPECIMENS

PART IV COPPER AND ITS ALLOYS AND THEIR EXAMINATION

0. FOREWORD

0.1 This Indian Standard (Part IV) was adopted by the Indian Standards Institution on 31 July 1975, after the draft finalized by the Metallography and Heat Treatment Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 The primary object of metallographic examination is to reveal the constituents and the structure of metals and their alloys by means of the microscope. In view of the diversity in available equipment, the wide variety of problems encountered and the personal element, this standard gives for the guidance of the metallographer only those practices which experience has shown are generally satisfactory.

0.3 This standard is being issued in parts covering general features, polishing, etching and examination of different metals. This part is fourth in the series and covers copper and its alloys. Other parts in this series are:

Part I General features
Part II Electrolytic polishing
Part III Aluminium and its alloys and their examination
Part V Iron and steel and their examination
Part VI Lead and its alloys and their examination
Part VII Magnesium and its alloys and their examination
Part VIII Nickel and its alloys and their examination
Part IX Precious metals and their examination
Part X Tin and its alloys and their examination
Part XI Zinc and its alloys and their examination

0.4 In the preparation of this standard assistance has been derived from ASTM E3-62 ' Standard methods of preparation of metallographic specimens ', issued by the American Society for Testing and Materials.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard (Part IV) covers the polishing, etching and examination of copper and its alloys.

2. POLISHING

2.0 For microscopic examination, secure a plane surface by filing, by rubbing over sheets of abrasive on a hard flat surface, or by use of an emery wheel copiously supplied with water.

2.1 Polishing is usually carried out in three stages, using wheel speeds varying over a wide range, depending on the alloy, abrasives, polishing cloth, and polisher's preference (speeds from 250 to 1 800 rev/min have been used). The first polishing wheel may be covered with sheets of abrasive paper of finenesses down to about No. 400. Usually, however, it is covered with canvas cloth to which is applied grit No. 500 silicon carbide, or No. 400 or finer grades of aluminium oxide. Cover the second wheel with wool broadcloth and use crushed sand stone as the abrasive. Cover the final wheel with either fine grade wool or a 'kitten's ear' broadcloth and employ a water suspension of aluminium oxide or finely powdered magnesium oxide as the abrasive.

2.2 Keep all polishing wheels wet during use by a water drip and keep the specimens, between steps, wet and thoroughly rinsed free of abrasives. After removal from the final wheel, the specimen may be immediately etched or rinsed in alcohol and quickly dried prior to etching. In much of the routine preparation of specimens, the final etching is depended upon to remove many shallow scratches.

2.3 Pure copper is more difficult to polish than its alloys, since a nearly perfect surface is required in order to detect the presence of cuprous oxide in the unetched specimen.

2.4 For macroscopic examination, the specimen may be prepared by grinding and rough polishing or by finishing with a fine tool on a shaper.

*Rules for rounding off numerical values (*revised*).

3. ETCHING

3.1 The etching reagents commonly recommended for copper and its alloys are given in Table 1.

3.2 The constituents of etching reagents specified in the above table should conform to the following Indian Standards:

Constituent	*Indian Standard*
Ammonium hydroxide	IS : 799-1955[1]
Distilled water	IS : 1070-1960[2]
Hydrogen peroxide	IS : 2080-1962[3]
Chromium trioxide	IS : 330-1968[4]
Hydrochloric acid	IS : 265-1962[5]
Nitric acid	IS : 264-1968[6]
Ferric chloride	IS : 711-1970[7]
Ethyl alcohol	IS : 321-1964[8]
Potassium permanganate	IS : 333-1969[9]
Sulphuric acid	IS : 266-1961[10]
Sodium chloride	IS : 4408-1967[11]
Ferrous sulphate	IS : 262-1967[12]
Sodium hydroxide	IS : 376-1969[13]
Acetic acid (glacial)	IS : 695-1967[14]

4. EXAMINATION AND IDENTIFICATION OF CONSTITUENTS

4.1 Combination of etches are frequently employed in the examination of copper alloys in order to secure contrast between several constituents. For instance, when alpha and beta structures are both present a ferric chloride etch following the ammonium hydroxide-hydrogen peroxide etch will darken the beta constituent. Similarly, a beautiful colouration of copper and phosphor-bronze may be obtained by following an ammonium hydroxide-hydrogen peroxide etch with a few seconds of electrolytic etching. In etching the copper-beryllium alloys, the specimen is frequently given a slight initial etch in potassium dichromate, followed by 10 to 15 seconds in the electrolytic solution.

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[1]Specification for ammonia, liquor, technical.
[2]Specification for water distilled quality (*revised*).
[3]Specification for stabilized hydrogen peroxide.
[4]Specification for chromium trioxide (*first revision*).
[5]Specification for hydrochloric acid (*revised*).
[6]Specification for nitric acid (*first revision*).
[7]Specification for ferric chloride, technical (*first revision*).
[8]Specification for absolute alcohol (*revised*).
[9]Specification for potassium permagnate (*first revision*).
[10]Specification for sulphuric acid (*revised*).
[11]Specification for sodium chloride, analytical reagent.
[12]Specification for ferrous sulphate, heptahydrote (*first revision*).
[13]Specification for sodium hydroxide, analytical reagent (*first revision*).
[14]Specification for acetic acid (*first revision*).

TABLE 1 ETCHING REAGENTS FOR COPPER AND ITS ALLOYS

(*Clause* 3.1)

Sl No.	Etching Reagent	Composition	Remarks	Use
(1)	(2)	(3)	(4)	(5)
1.	Ammonium hydroxide —hydrogen peroxide	NH_4OH 5 parts H_2O 5 parts H_2O_2 (3 percent) 2 to 5 parts	Peroxide content varies directly with copper content of alloy to be etched. Immersion or swabbing for about 1 minute. Fresh H_2O_2 is desirable for good results	Generally used for copper and many of its alloys. Film on etched aluminium bronze removed by weak Grard's solution
2.	Ammonium hydroxide	Diluted solutions	Immersion	Polish-attack etching of brass and bronze
3.	Ammonium hydroxide-ammonium persulphate	NH_4OH 1 part H_2O 1 part $(NH_4)_2S_2O_8$ 2 parts (2·5 percent)	Immersion	Polish-attack of copper and some alloys
4.	Ammonium persulphate	$(NH_4)_2S_2O_8$ 10 g H_2O 90 ml	Use either cold or boiling. Immersion	Copper, brass, bronze, nickel silver, aluminium bronze
5.	Chromic acid	Saturated aqueous solution (CrO_3)	Immersion or swabbing	Copper, brass, bronze, nickel silver (plain etch)
6.	Chromic acid	1 percent aqueous solution (CrO_3)	Use electrolytically at 6 V, with aluminium cathode, for 3 to 6 s	Aluminium bronze and beryllium copper
7.	Chromic acid-hydrochloric acid	CrO_3 (10 to 15 percent) 50 ml HCl 1 to 2 drops	Add HCl at time of use immersion	Same as reagent No. 5. Colour by electrolytic etching or $FeCl_3$ reagents

8. Chromic acid-nitric acid	HNO_3 50 ml CrO_3 20 g H_2O 30 ml or HNO_3 5 ml CrO_3 20 g H_2O 75 ml		Immersion	Aluminium bronze; film from polishing removed by 10 percent HF
9. Copper ammonium chloride-ammonium hydroxide	10 percent aqueous solution of copper ammonium chloride plus NH_4OH to neutrality or alkalinity		Immersion. Wash specimen thoroughly	Best for darkening large areas of beta in alpha-beta brass, copper, brass, nickel silver
10. Ferric chloride	$FeCl_3$ 5 19 5 25 1 8 10 3 HCl 50 6 10 25 20 25 1 10 H_2O 100 100 100 100 100 100 100 100		Immersion or swabbing. Etch lightly or by successive light etches to required results	Copper, brass, bronze, aluminium bronze; darkens beta in brass; gives contrast following dichromate and other etches
11. Ferric chloride	$FeCl_3$ 5 g Ethyl alcohol 96 ml HCl 2 ml		Immersion or swabbing for 1 second to several minutes	Copper, aluminium, magnesium nickel and zinc alloys, etc
12. Nitric acid	Various concentrations		Immersion or swabbing, $AgNO_3$ (0·15 to 0·3 percent) added to 1 : 1 nitric acid solution gives a brilliant deep etch	Deep etching
13. Potassium dichromate	$K_2Cr_2O_7$ 2 g H_2SO_4 8 ml NaCl (saturated solution) 4 ml H_2O 100 ml		NaCl can be replaced by 1 drop of HCl to 25 ml solution added just before using immersion	Copper; copper alloys of beryllium manganese, and silicon; nickel silver, bronze; and chromium copper. Followed by $FeCl_3$ or other contrast etch

(*Continued*)

TABLE 1 ETCHING REAGENTS FOR COPPER AND ITS ALLOYS — *Contd*

Sl. No.	Etching Reagent	Composition	Remarks	Use
(1)	(2)	(3)	(4)	(5)
14.	Electrolytic etch	$FeSO_4$ 30 g NaOH 4 g H_2SO_4 100 ml H_2O 1 900 ml	Use 0·1 A at 8 to 10 V. Generally not over 15 s. Do not swab surface after etching	Darkens beta in brass, gives contrast after H_2O_2 etch. Nickel silver bronze and other alloys
15.	Electrolytic etch	HNO_3 10 ml Glacial acetic Acid 5 ml H_2O 85 ml		Is very satisfactory for etching high-nickel alloys such as 20 to 30 percent cupro-nickel and Monel. It tends to minimize the striations which appear after etching due to coring effect

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